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## Free-Radical Solution-Polymerization of Trifluoronitrosomethane with Tetrafluoroethylene

The effect of including free-radical initiators in solution-polymerizations of trifluoronitrosomethane ( $\text{CF}_3\text{NO}$ ) with tetrafluoroethylene ( $\text{C}_2\text{F}_4$ ) was investigated to determine if more than an equimolar quantity of  $\text{C}_2\text{F}_4$  could be incorporated in the copolymer.

The polymerization initiators selected for study were methylethyl ketone peroxide, *t*-butyl hydroperoxide, and isopropylperoxydicarbonate; methylene chloride was selected as solvent and the temperature schedule for the polymerization was set by preliminary experiments at 5 days at  $-35^\circ\text{C}$  and 2 days at  $0^\circ\text{C}$ . The free-radical initiator was used at the rate of 0.1 g for each 0.20 mole of  $\text{CF}_3\text{NO}$ . In each case, a 50-percent excess of  $\text{C}_2\text{F}_4$  was charged into the reaction vessel.

Polymer conversions were good, and in some instances experimental data suggested that the  $\text{C}_2\text{F}_4$  content exceeded the theoretical amount for a 1 : 1 copolymer. The products were elastomeric gums which exhibited a very low order of flow; however, they were not completely soluble in the FC-43 solvent used for measurement of relative viscosity. Hexafluorobenzene also did not dissolve the copolymer completely.

For polymerization, a 500-ml heavy-walled glass reactor (equipped with aerosol-compatible couplings and needle valve) was charged with 100 g methylene chloride, followed by 0.1 g of the free-radical initiator. The solvent was degassed three times by a freeze-thaw technique at liquid nitrogen temperature. Then

20.0 g (0.2 mole)  $\text{CF}_3\text{NO}$  and 32.0 g (0.3 mole)  $\text{C}_2\text{F}_4$  were charged successively into the polymerization vessel while it was immersed in liquid nitrogen. Transfers were made by expansion of the vaporized monomers into a calibrated volume at a measured pressure followed by liquefaction at liquid nitrogen temperature. The sealed vessel (shaken periodically) was maintained in a refrigerated bath at  $-35^\circ\text{C}$  for a 5-day period and then for 2 days at  $0^\circ\text{C}$ . The cooled vessel was vented and allowed to come to room temperature while its contents were kept under a nitrogen atmosphere. The system was evacuated to remove residual monomer, solvent, and other volatiles. The remaining gum was heated for 4 hours at  $60^\circ\text{C}$  in nitrogen at low pressure.

### Note:

Requests for further information may be directed to:  
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### Patent status:

No patent action is contemplated by NASA.

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